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A FUNDAMENTAL STUDY OF FLOW AND FRACTURE IN BERYLLIUM.(U)
JAN 78 D WEBSTER, D D CROOKS DAAG29-76-C-0051

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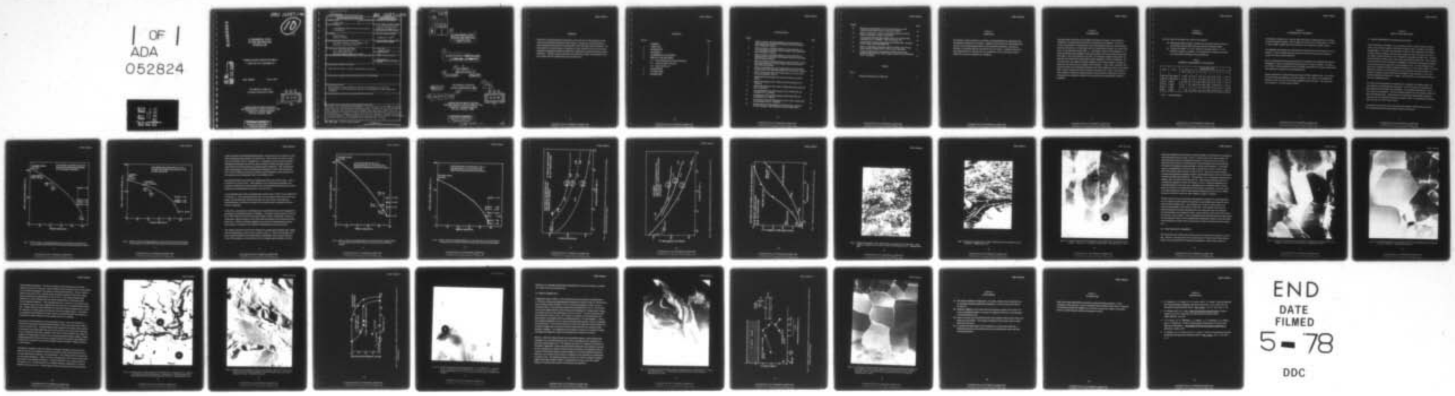

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INTERIM TECHNICAL REPORT FOR PERIOD

1 JUNE 1976 TO 31 DECEMBER 1977

LMSC-D566852 January 1978

ARO PROJECT P-13387-MS

CONTRACT DAAG29-76-C-0051

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ARO 13387.1-MS

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER 13387.1-MS	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) A Fundamental Study of Flow and Fracture in Beryllium		5. TYPE OF REPORT & PERIOD COVERED Interim Technical Report 1 Jun 76 - 31 Dec 77
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) Donald Webster, Donald D. Crooks		8. CONTRACT OR GRANT NUMBER(s) DAAG29 76 C 0051 new
9. PERFORMING ORGANIZATION NAME AND ADDRESS Lockheed Missiles & Space Company, Inc. Palo Alto, California 94304		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
11. CONTROLLING OFFICE NAME AND ADDRESS U. S. Army Research Office Post Office Box 12211 Research Triangle Park, NC 27709		12. REPORT DATE January 1978
		13. NUMBER OF PAGES 31
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		15. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

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19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

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The effect of thermomechanical treatments on the grain size of HIP beryllium powder block and cast beryllium alloys has been studied. A grain size controlled fracture mode change has been observed in both powder source and ingot source beryllium. The mechanism of recrystallization in beryllium has been observed to be the in situ transformation of subgrains to grains by dislocation migration from grain interiors to grain boundaries. Maximum impact resistance was observed in partly recrystallized structures rather than the expected fully recrystallized state.

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1 JUN[redacted]76 [redacted]31 DEC[redacted]77

by

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Donald Webster
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ARO PROJECT P-13387-MS
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SUMMARY

The effect of thermomechanical treatments on the grain size of HIP beyllium powder block and cast beryllium alloys has been studied. A grain size controlled fracture mode change has been observed in both powder source and ingot source beryllium. The mechanism of recrystallization in beryllium has been observed to be the in situ transformation of subgrains to grains by dislocation migration from grain interiors to grain boundaries. Maximum impact resistance was observed in partly recrystallized structures rather than the expected fully recrystallized state.

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Section 1
OBJECTIVE

The objective of this program is to develop an understanding of the fundamental flow and fracture mechanism in beryllium. Emphasis will be placed on understanding the reasons for the marked improvement in the mechanical properties, particularly ductility, of beryllium that has occurred in the last 5 years. A specific objective of this program will be to indicate how new mechanisms of flow and fracture can be utilized to overcome beryllium's low toughness.

Section 2
BACKGROUND

Although modern beryllium has the highest structural efficiency of any material or composite, its use has been restricted by low ductility and toughness. In the last 5 years, the ductility of beryllium has been dramatically increased so that 6% elongation in all directions of a semicommercial, hot isostatically pressed, low oxide (0.5%) block can be produced routinely (Ref. 1). Still higher values of 13% elongation in virtually nontextured upset forgings of the same material have been produced experimentally (Ref. 2) and recent Russian work (Ref. 3) using upset forging of high purity cast material has enabled three-dimensional tensile elongation of 22% to be obtained. For most structure, this degree of tensile elongation is more than adequate to permit designs that can utilize the full strength of the beryllium. At this stage, beryllium compares favorably in its mechanical properties with other engineering alloys in all aspects other than its resistance to impact shocks in the presence of a stress concentration. This condition is simulated experimentally by charpy impact testing which is used on this program to evaluate the effects of microstructural and mechanistic changes on toughness.

Section 3
MATERIALS

Two basic types of beryllium were used on this program:

(1) High purity powder products formed from electrolytic flake and then hot isostatically pressed (HIP) by Kawecki Berylco Industries (KBI)

(2) Vacuum cast ingots 2- to 3-in. diameter containing small (~ 0.1%) addition of titanium, vanadium, or chromium plus a high purity vacuum cast ingot (EFI) containing no alloying additions

Table 1
CHEMICAL COMPOSITION OF MATERIALS

Alloy	Form	Composition PPM								
		BeO	Mg	Si	Al	Fe	C	Ti	Ni	Cr
BOP 32	HIP Block	7,000	35	140	33	178	210	N.D.	250	N.D.
BOP 30	HIP Block	6,700	41	120	35	185	280	N.D.	265	N.D.
PS 20	Sheet	10,700	100	90	480	950	620	N.D.	N.D.	N.D.
Be-Ti	Ingot	N.D.	20	380	670	290	520	1,900	N.D.	N.D.
Be-Cr	Ingot	N.D.	10	70	20	140	300	N.D.	N.D.	1,300
EFI	Ingot	40	10	100	90	110	290	N.D.	N.D.	N.D.

N.D. = Not determined.

Section 4
EXPERIMENTAL TECHNIQUE

All beryllium samples were canned in mild steel prior to forging extrusion or rolling to prevent surface cracking. Upset forging was carried out at 1033K (1400 F), rolling at either 1033K or 922K (1200 F) and extrusion (10:1 reduction) at 894K (1150F).

Fractographic examination was performed by scanning electron microscopy for coarse grained ($> 50\ \mu m$) samples and by transmission electron microscopy of shadowed replicas for finer grained materials.

Direct transmission electron microscopy of the beryllium was conducted after electropolishing in a solution containing 82% ethylene glycol, 5% H_2O, 9% HNO_3, 2% H_2SO_4, and 2% HCl. Both conventional (120 kV) and high-voltage (650 kV) electron microscopes were utilized to examine the electropolished foils.

Charpy specimens for impact testing were made with the rounded notch, 1-mm radius and 2-mm deep conventionally used with beryllium. The surface was etched before testing to remove 0.1 mm of potentially damaged surface material. The specimens were tested on a 24 ft/lb impact machine.

Section 5
RESULTS AND DISCUSSION

5.1 GRAIN REFINEMENT BY RECRYSTALLIZATION

Grain refinement in beryllium, as in most other metals, confers property improvements such as higher toughness and tensile elongation (Ref. 1) together with higher strength (Ref. 4), but there are limitations to how far this refinement can be taken. In powder block beryllium, the grain size is similar to that of the input powder and any degree of grain refinement can be obtained by reducing the powder size. However, this increases the surface area of the powder and hence the oxide level. This counteracts the benefits of grain refinement on toughness and ductility and optimum properties are observed when the grain size is approximately 10 μm. In cast beryllium where oxide is not normally present in sufficient quantities to act as a grain refiner, it is very difficult to produce a recrystallized grain size less than 30 μm even in sheet. An alternative method of grain refinement investigated in this program is to use intermetallic compounds of titanium, vanadium, and chromium to act as grain refiners in cast and wrought beryllium.

Two types of thermomechanical treatment were conducted to determine which was most effective in producing grain refinement. In the first type, the beryllium was deformed about 20% and fully recrystallized at the minimum temperature at which this could be accomplished. The recrystallized material was then rolled to about 40% and recrystallized again. This process was repeated for 90% and 96% reductions. The grain size was reduced at each step as shown in Fig. 1 for high-purity beryllium (EFI) and Fig. 2 for the 0.19% Ti alloy.

The initial "as-cast" grain size was taken as the dendrite width which is about ten times smaller than the average dendrite length in both cases.

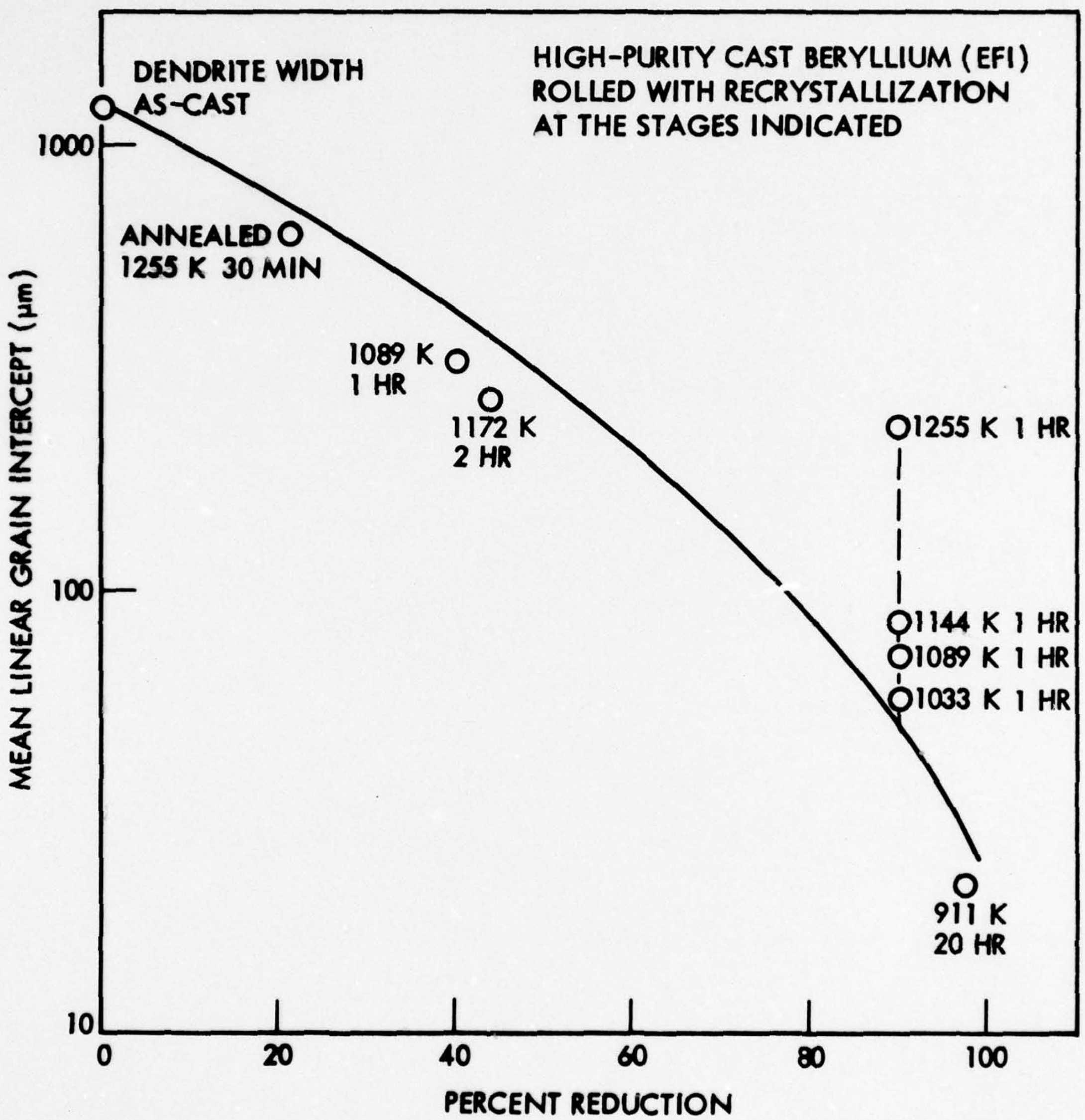

Fig. 1 Effect of Degree of Rolling Reduction on the Grain Size of High-Purity Cast Beryllium (EFI) Rolled With Intermediate Recrystallization Anneals

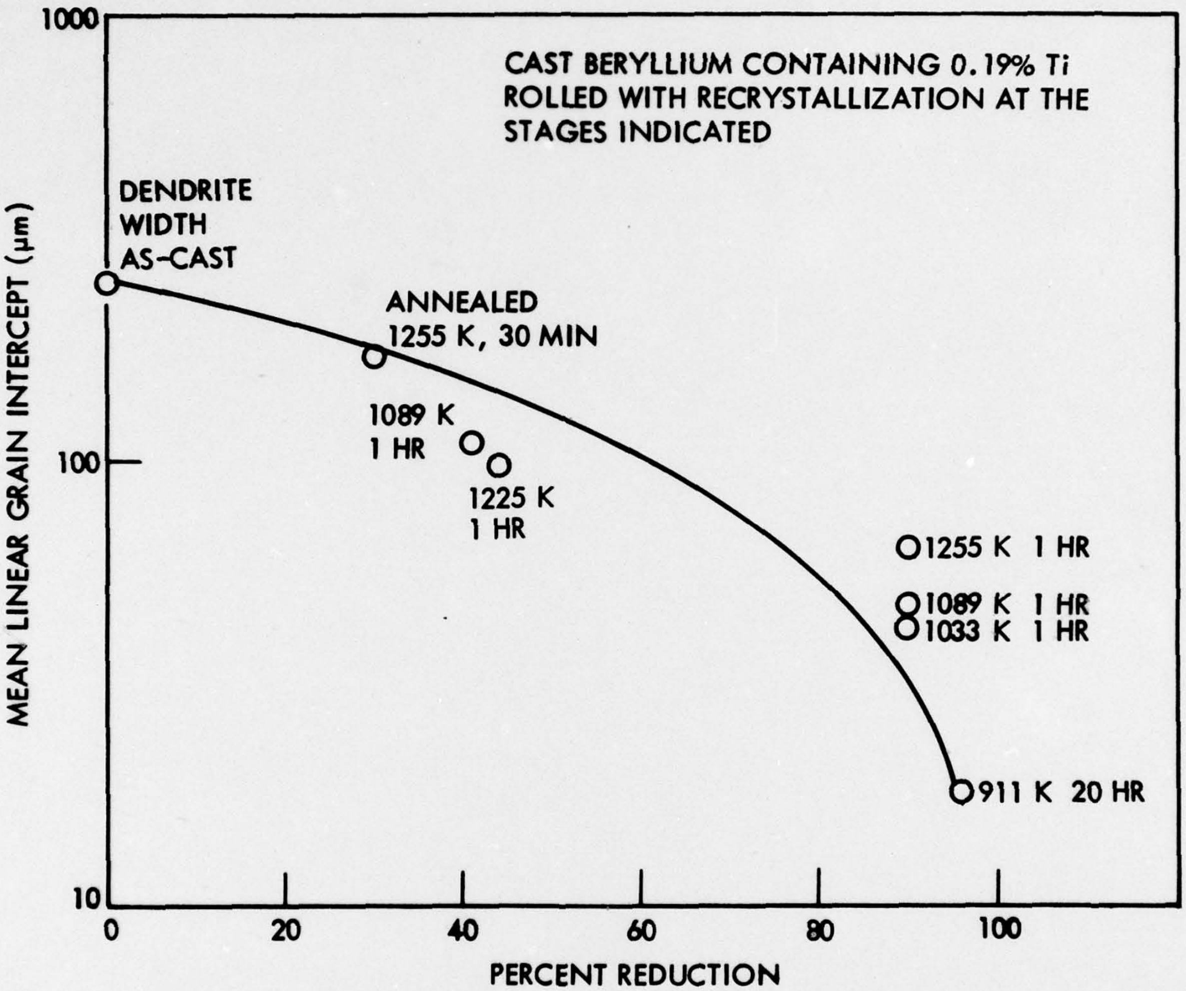

Fig. 2 Effect of Degree of Rolling Reduction on the Grain Size of Cast Beryllium Containing 0.13% Cr Rolled With Intermediate Recrystallization Anneals

In the second type of thermomechanical process, the specimens were rolled 90 or 96% before recrystallization anneals were performed. These results are shown in Figs. 3 and 4 for EFI and 0.19% Ti, respectively. A comparison of the two types of thermomechanical treatments shows that the direct reduction without interstage anneals is more effective for both materials in producing grain refinement. This result follows from an examination of the recrystallization mechanism as will be discussed below. The 0.19% Ti addition can be seen to produce grain refinement in both processes and also produces some refinement of the as-cast dendrite size.

Recrystallized grain sizes after upset forging and rolling are shown in Figs. 5 and 6 for EFI and a 0.13% Cr alloy. There appears to be no particular advantage to any particular temperature in terms of grain size, but annealing temperatures above 950 K produce fully recrystallized structures in less than 72 hr.

A more detailed study of the recrystallization of EFI and Be-0.13% Cr were conducted at 977 K (Fig. 7). The recrystallized grain size was measured in both alloys in the early stages of recrystallization when the structure was only partially recrystallized. Recrystallization is obviously retarded in the chromium alloy, probably by the intermetallic compounds.

In this alloy, grain growth is continuing in the recrystallized areas even though further nucleation of recrystallized grains is inhibited. The optical microstructure of EFI and Be 0.13% Cr after 5 min at 977 K is shown in Figs. 8 and 9, respectively. In both materials faint, ghostly "grains" can be seen inside what appears to be large unrecrystallized areas. These are actually subgrains in the process of transforming to grains. This process is revealed in more detail by transmission electron microscopy.

The subgrain structure of EFI in the vicinity of an original grain boundary after rolling 96% and annealing for 1 min at 977 K is shown in Fig. 10. Most of the subrains have poorly defined boundaries and a high density of dislocations in the subrain interiors. A few of the subgrains, such as those at A on the original grain boundary, have low

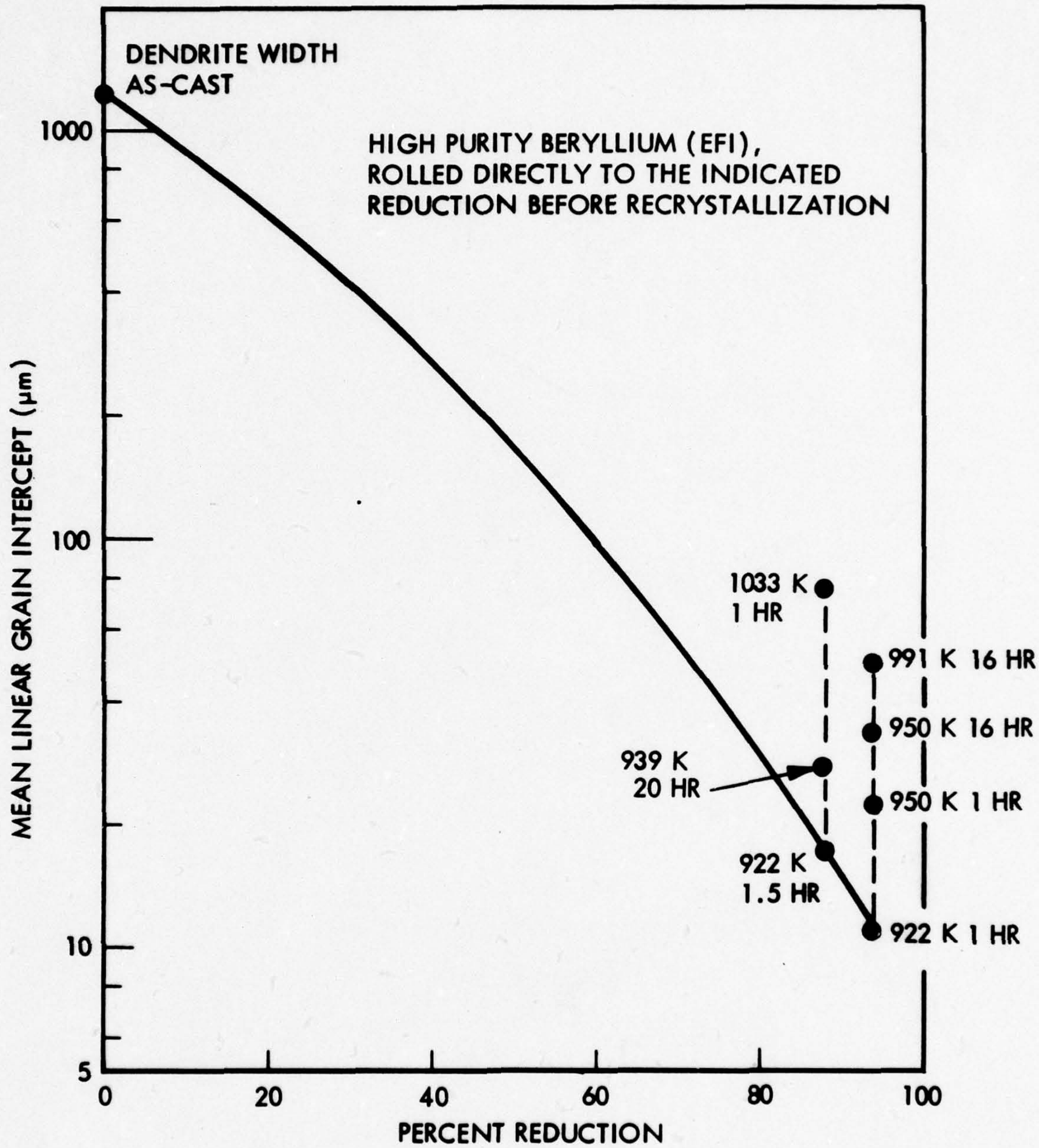

Fig. 3 Effect of Degree of Rolling Reduction on the Grain Size of High-Purity Cast Beryllium (EFI) Rolled Without Intermediate Recrystallizing Anneals

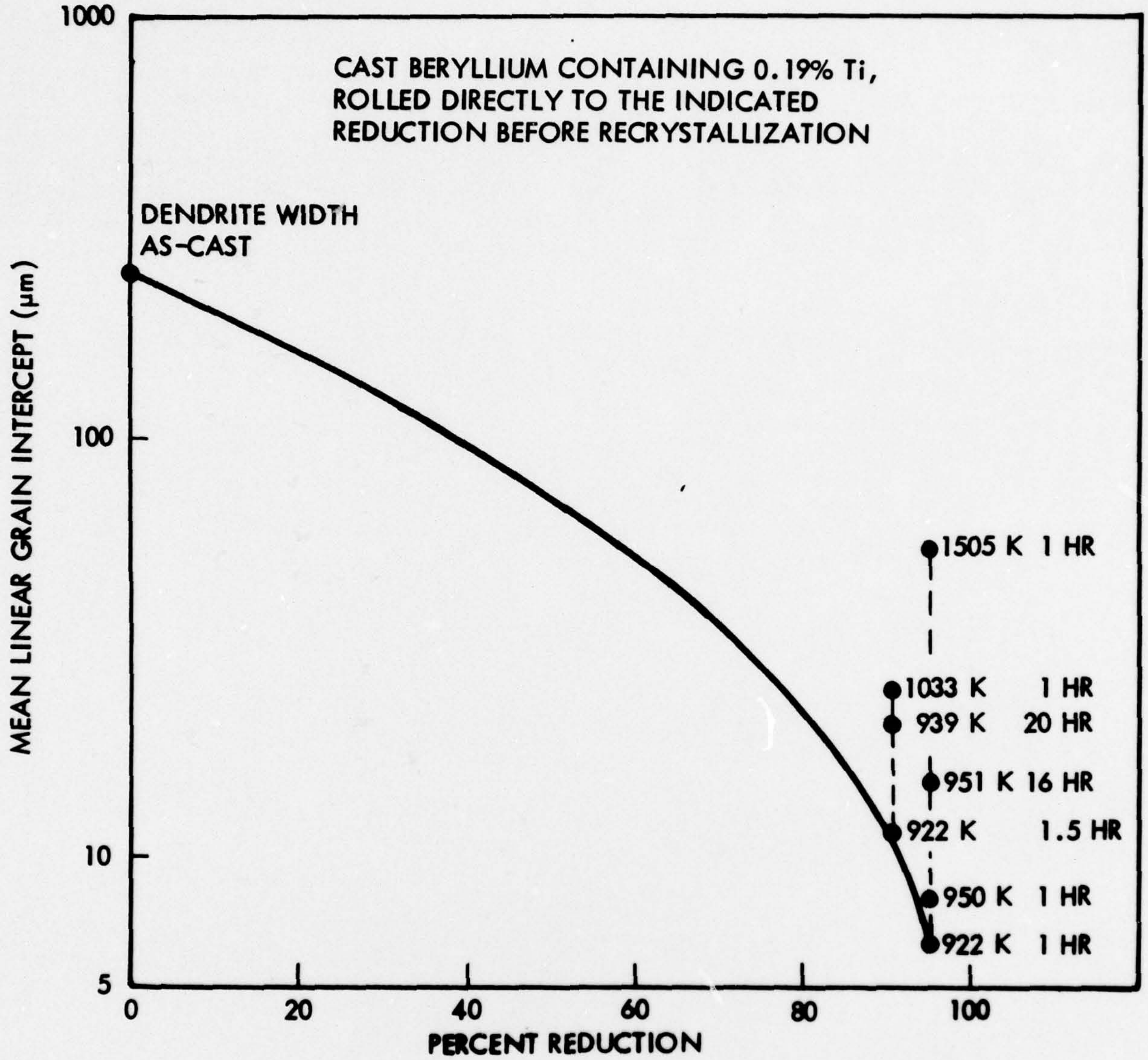

Fig. 4 Effect of Degree of Rolling Reduction on the Grain Size of Cast Beryllium Containing 0.19% Ti, Rolled Without Intermediate Recrystallizing Anneals

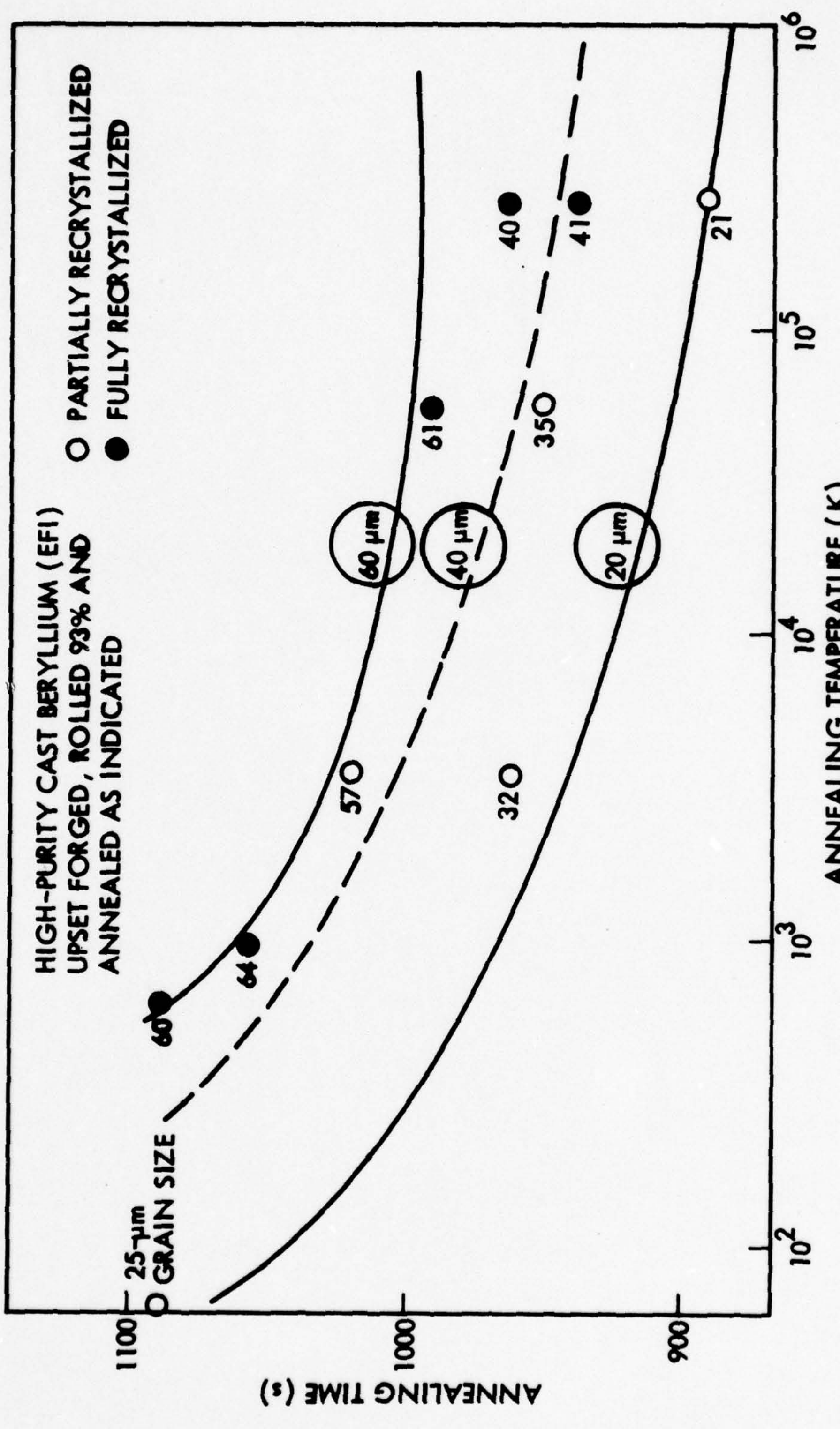

Fig. 5 Effect of Annealing Time and Temperature on the Grain Size of High-Purity Cast Beryllium Rolled 93% After Upset Forging

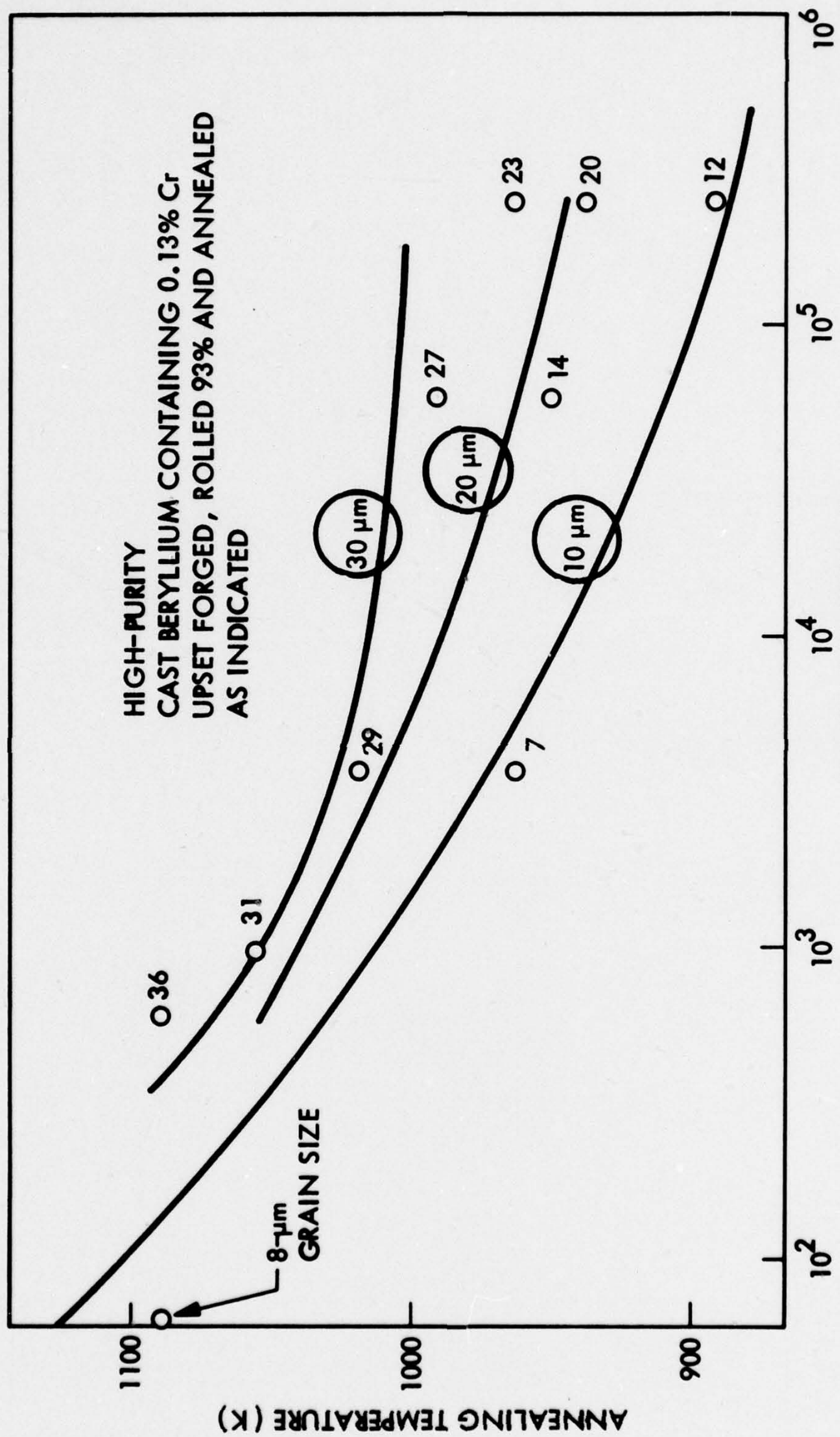

Fig. 6 Effect of Annealing Time and Temperature on the Grain Size of Cast Beryllium 0.13% Cr

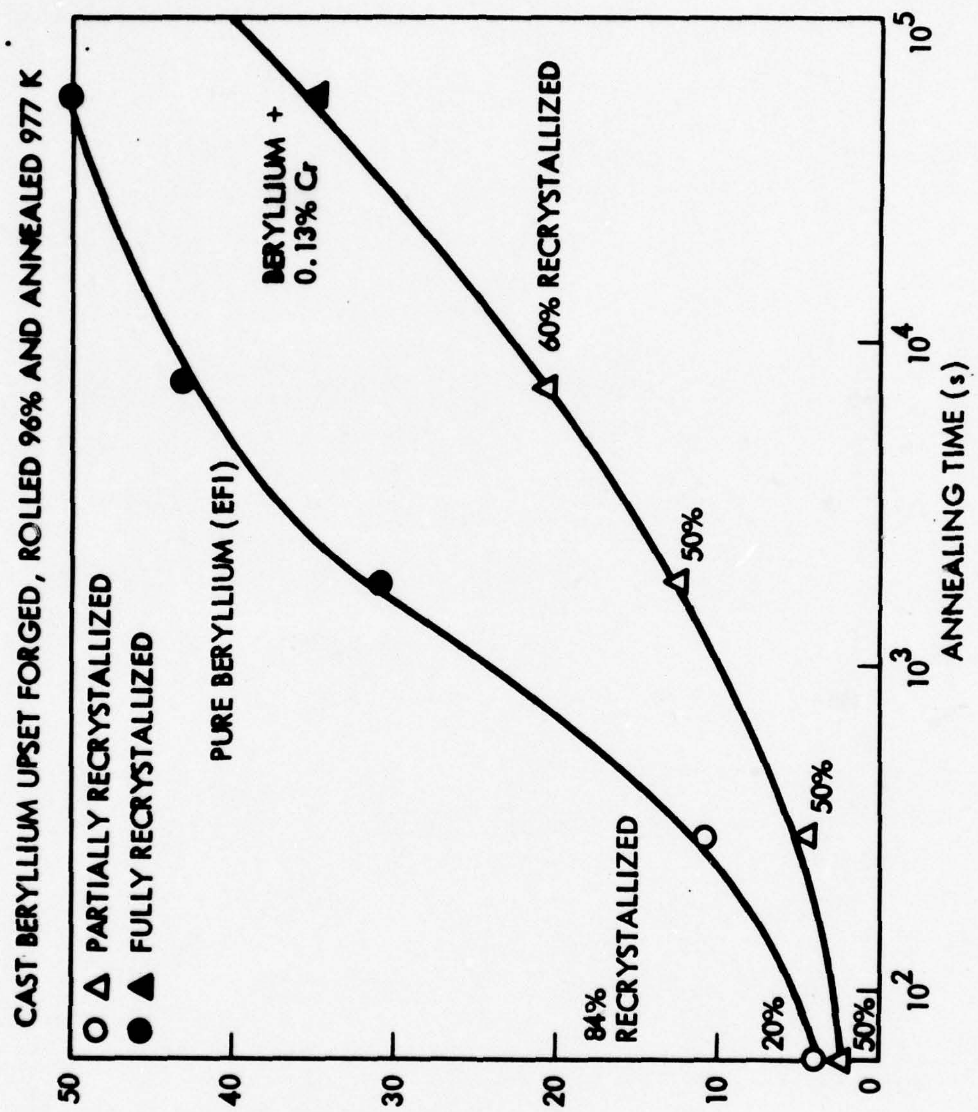

Fig. 7 Isothermal Grain Growth at 977 K for EFI and Be 0.13% Cr Alloys

Fig. 8 Optical Micrograph of EFI, Rolled 96% and Annealed 977 K 5 Minutes. Faint 'ghostly' subgrains can be seen in some unrecrystallized areas. Magnification 200×

Fig. 9 Optical Micrograph of Be 0.13% Cr Rolled 96% and Annealed at 977 K 5 Minutes. Magnification 200×

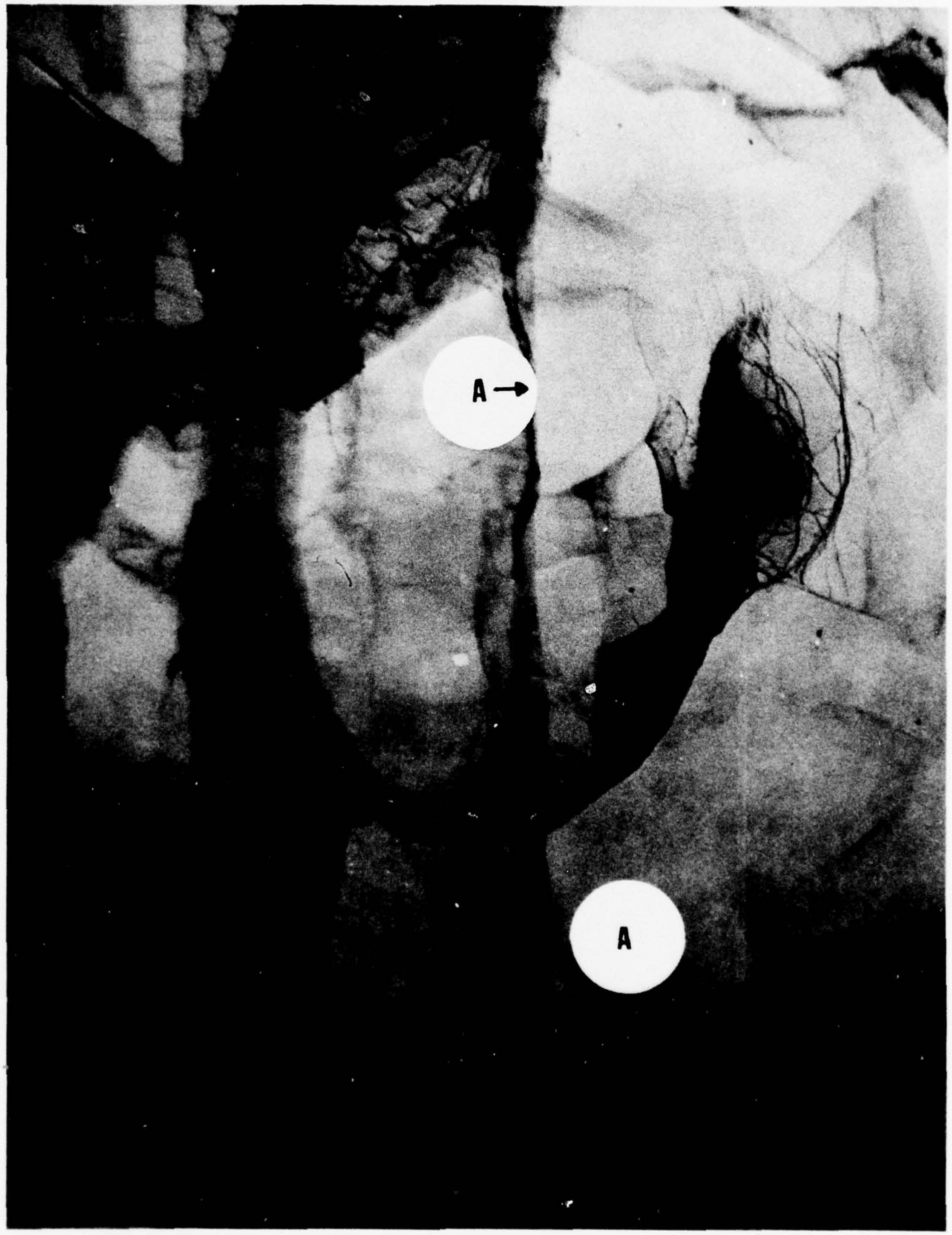

Fig. 10 Transmission Electron Micrograph of EFI, Rolled 96% and Annealed 977 K, 1 Minute. Grains at A are almost recrystallized. Magnification 16,000×

dislocation densities and may appear in polarized light to be the ghostly recrystallized grains described above for Figs. 8 and 9. In other areas of the same sample, the recrystallization process has progressed further as shown for grain A in Fig. 11 where the grain interior is dislocation free and the boundaries are sharply defined. This grain would appear in polarized light to be fully recrystallized. A still later stage of recrystallization is shown in Fig. 12 which shows the Be 0.13% Cr alloy after 90% reduction and an anneal of 5 min at 977 K. This area shows a boundary between unrecrystallized subgrains and fully recrystallized grains with low dislocation densities and high angle boundaries. The particle at A is a beryllium-chromium intermetallic with an associated deformation void. With continued annealing, the unrecrystallized subgrains in all the cast alloys are seen to transform gradually to recrystallized grains of the same size, although there is no definite point at which a grain can be said to be recrystallized by microscopic observation. However, there is a sharp transition in fracture mode during annealing which can be used to define the point at which the conversion from subgrain to grain is complete.

It follows from the above observation indicating the mechanism of recrystallization to be by subgrain transformation that the finest recrystallized grain size will result from the finest subgrain size. This subgrain size is found in most metals to be inversely proportional to the amount of cold work. This explains why in the two thermomechanical processes described above the finest grain size was produced when intermediate recrystallization steps, which would have reduced the total amount of cold work, were avoided. If the recrystallization mechanism had been by the growth of grain boundary nuclei, then the gradual grain refinement produced by the intermediate recrystallization steps in the second process would have produced the finer grain size.

5.2 FRACTURE MODE TRANSITION

The normal fracture mode in both powder source and ingot source beryllium is cleavage. However, when the grain size is reduced below a certain critical value characteristic of each composition and method of manufacture, the fracture mode is by

Fig. 11 Transmission Electron Micrograph of EFI Rolled 96% and Annealed 977 K, 1 Minute. Grain at A is recrystallized. Magnification 8000×

Fig. 12 Transmission Electron Micrograph of Be 0.13% Cr Rolled 90% and Annealed at 977 K, 5 Minutes. Magnification 16,000×

grain boundary separation. Previous work (Ref. 2) has indicated grain boundary sliding occurs prior to fracture, and this additional slip mechanism may be responsible for the improved toughness and ductility of fine-grained materials. The two fracture modes can be seen in Fig. 13 which shows the fracture surface of EFI after rolling 96% and partially recrystallizing for 5 min at 977 K. The unrecrystallized area, typified by A, shows cleavage with only minor perturbations in the fracture path across subgrain boundaries. The recrystallized areas, such as B, have grains which are about the same size as the subgrains but where fracture is completely intercrystalline. Similar fracture mode transitions are observed in powder products (Fig. 14) but the transition is at finer grain sizes than for cast and wrought products. A partially recrystallized sheet (PS20) shows bands of fine recrystallized grains fracturing in an intergranular manner while the coarser grains fracture by cleavage.

The fracture mode transition is shown graphically in Fig. 15 for cast beryllium in textured and nontextured wrought products together with hot-pressed powder products of various oxide levels. Texturing displaces the transition of the ingot source product to finer grain sizes while high oxide levels do the same for the hot-pressed materials. It may be significant that these variables which tend to reduce ductility also reduce the tendency for intergranular fracture with its associated grain boundary sliding. Higher testing temperatures which are known to rapidly improve the ductility and toughness of beryllium are expected to increase the tendency for grain boundary sliding as they do in other metals.

Preliminary attempts to study the initiation of the grain boundary cracks have been made by the use of transmission electron microscopy to study fine-grained sheet that has been deformed by rolling at room temperature. Very sharp cracks were observed on some grain boundaries. Figure 16 shows a fine grained 0.19% Ti alloy where all the grains have a fine dislocation network. A sharp crack about 1-μm long is visible on one boundary at A. Small particles of a beryllium-titanium compound 60- to 150-nm diameter can also be seen. A much longer but still very sharp crack was observed in

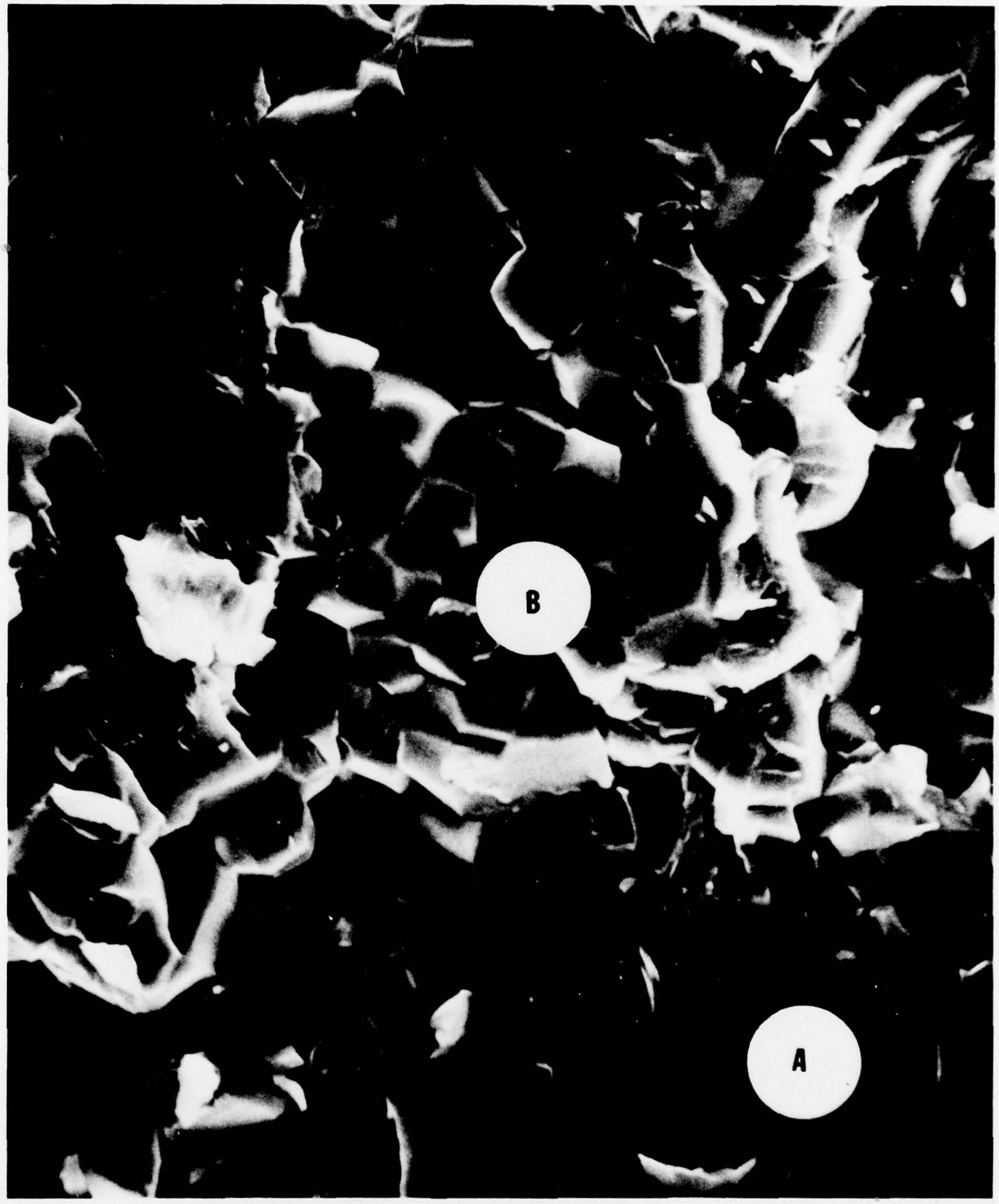

Fig. 13 Scanning Electron Micrograph of EFI Rolled 96%, Annealed 977 K, 5 Minutes and Fractured at Room Temperature. Subgrains at A Show Cleavage Fracture While Grains at B Show Intergranular Fracture. Magnification 1320×

Fig. 14 Shadowed Carbon Replica of the Fracture Surface of PS 20 Powder Source Sheet in the As-Received Condition. Small fine grain areas show intergranular fracture. Magnification 8000×

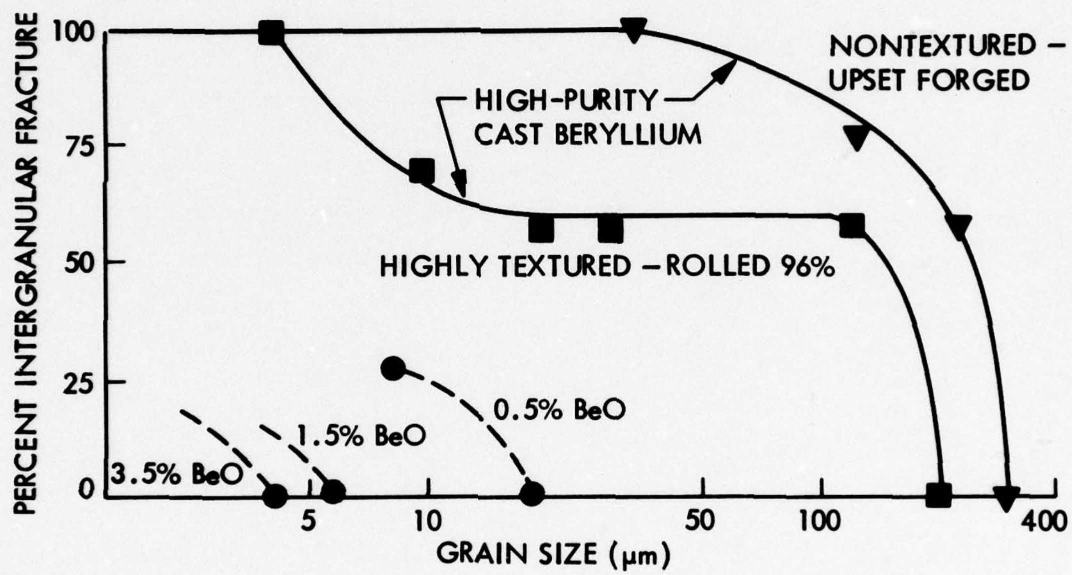

Fig. 15 Effect of Grain Size, Texture and Oxide Content on Fracture Mode on Beryllium at Room Temperature

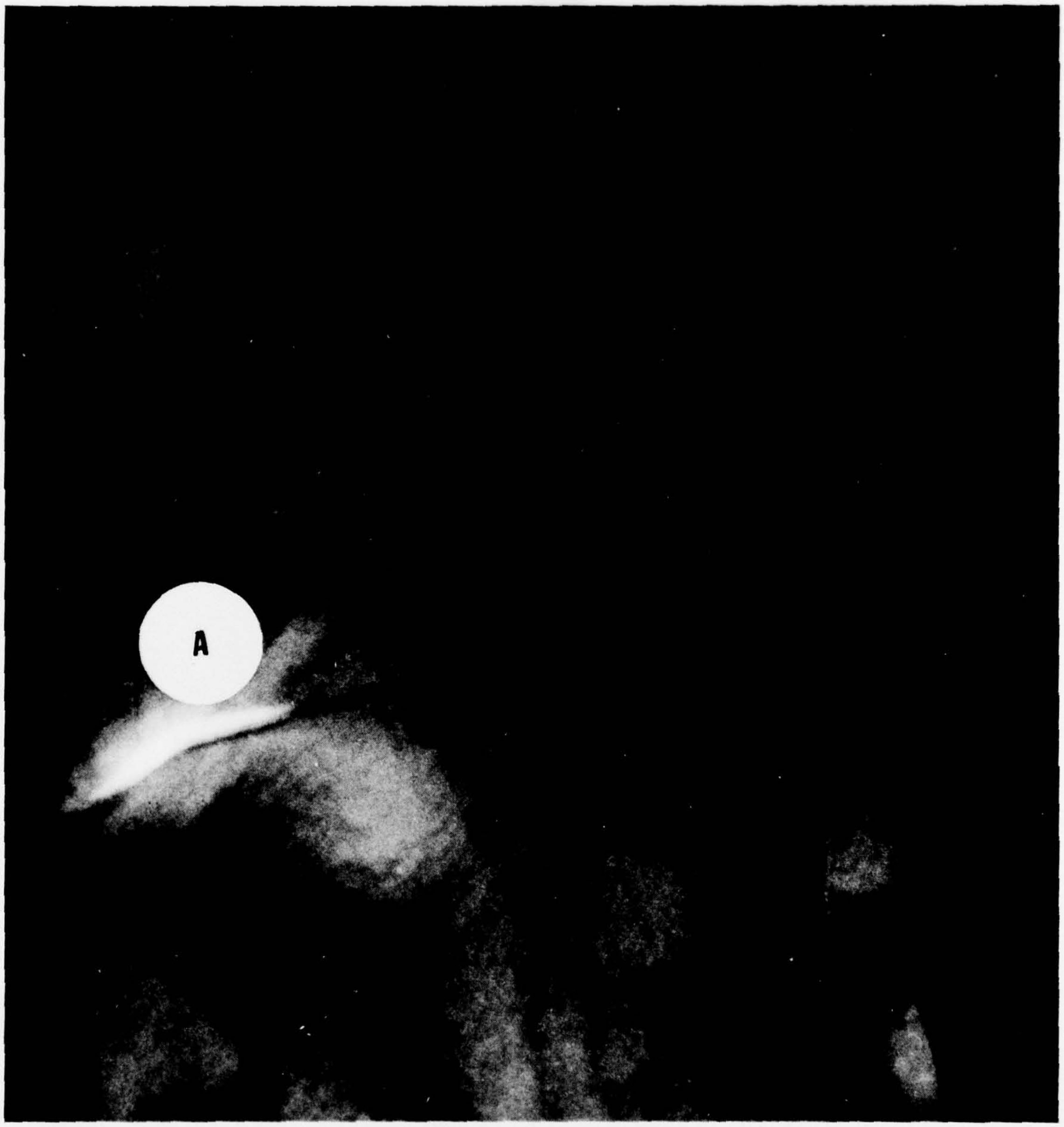

Fig. 16 Transmission Electron Micrograph of Be 0.1% Ti Rolled 96%, Annealed 977 K, 10 Minutes Then Cold Rolled 15%. A small crack has appeared along a grain boundary at A as a result of the cold rolling. Magnification 30,000×

EFI (Fig. 17), although the dislocation distribution was much less uniform, probably as a result of the coarser grain size.

5.3 IMPACT RESISTANCE

Preliminary impact results on hot isostatically pressed beryllium containing about 0.7% BeO for various thermomechanical treatments are shown in Fig. 18. These alloys have an impact energy of about 1 J is the as-pressed condition, but this can be increased to about 3 J by extrusion and partial recrystallization at 977 K. If the material is upset forged and then rolled without recrystallizing, the charpy impact energy is raised to about 6 J after annealing at 977 K for 10 min. The microstructure after this treatment is shown in Fig. 19 to consist of a mixture of unrecrystallized subgrains about 1-μm diameter and recrystallized grains 2- to 4-μm diameter. The unrecrystallized subgrains occur in areas containing oxide particles which are inhibiting recrystallization. It can be seen from Fig. 18 that the forged and rolled beryllium has an impact energy close to that of Al 7075-T6 tested with the same specimen under identical conditions. It is significant that the impact resistance is reduced by further annealing even though the extent of recrystallization continues to increase.

However, while the volume fraction of recrystallized regions is increasing during annealing, the recrystallized grain size is also increasing and this apparently is a powerful compensating factor. The optimum structure for toughness and ductility in beryllium is apparently not the long expected fine grain fully recrystallized structure but one consisting of a relatively small fraction of ultrafine recrystallized grains in a matrix of what would appear in polarized light to be an unrecrystallized structure. However, this structure would consist of subgrains on the threshold of transforming to grains in situ. The optimum properties would occur when the subgrain boundary angle became high enough to deflect a transgranular cleavage crack into a grain boundary so that the local fracture would become intergranular.

Fig. 17 Transmission Electron Micrograph of EFI Rolled 96%, Annealed 977 K, 1 Hour and Cold Rolled 14%. A sharp grain boundary crack has been initiated. Magnification 20,000×

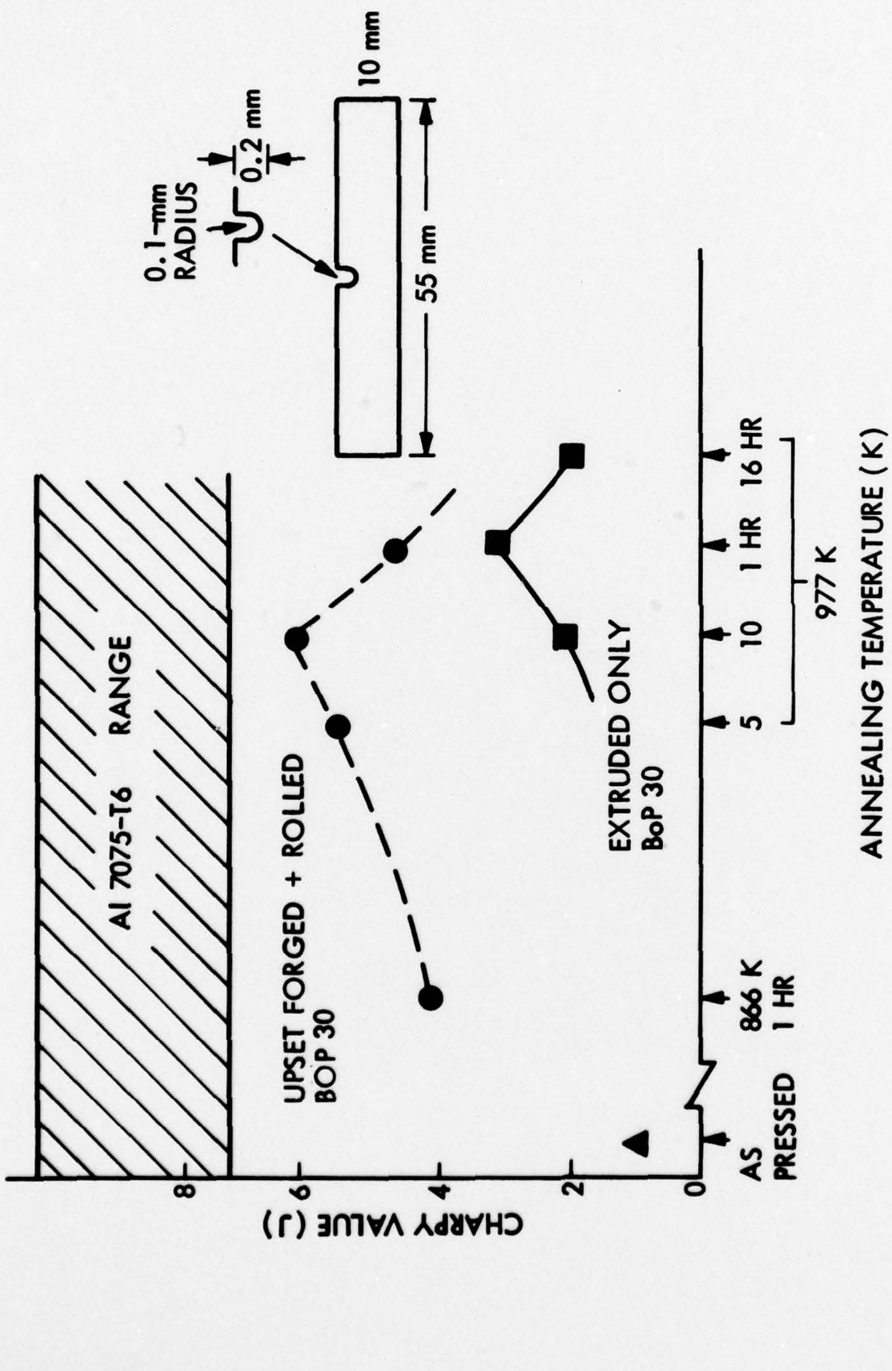

Fig. 18 Effect of Annealing Treatments and Hot Working on the Charpy Impact Energy of High-Purity Powder Source Beryllium

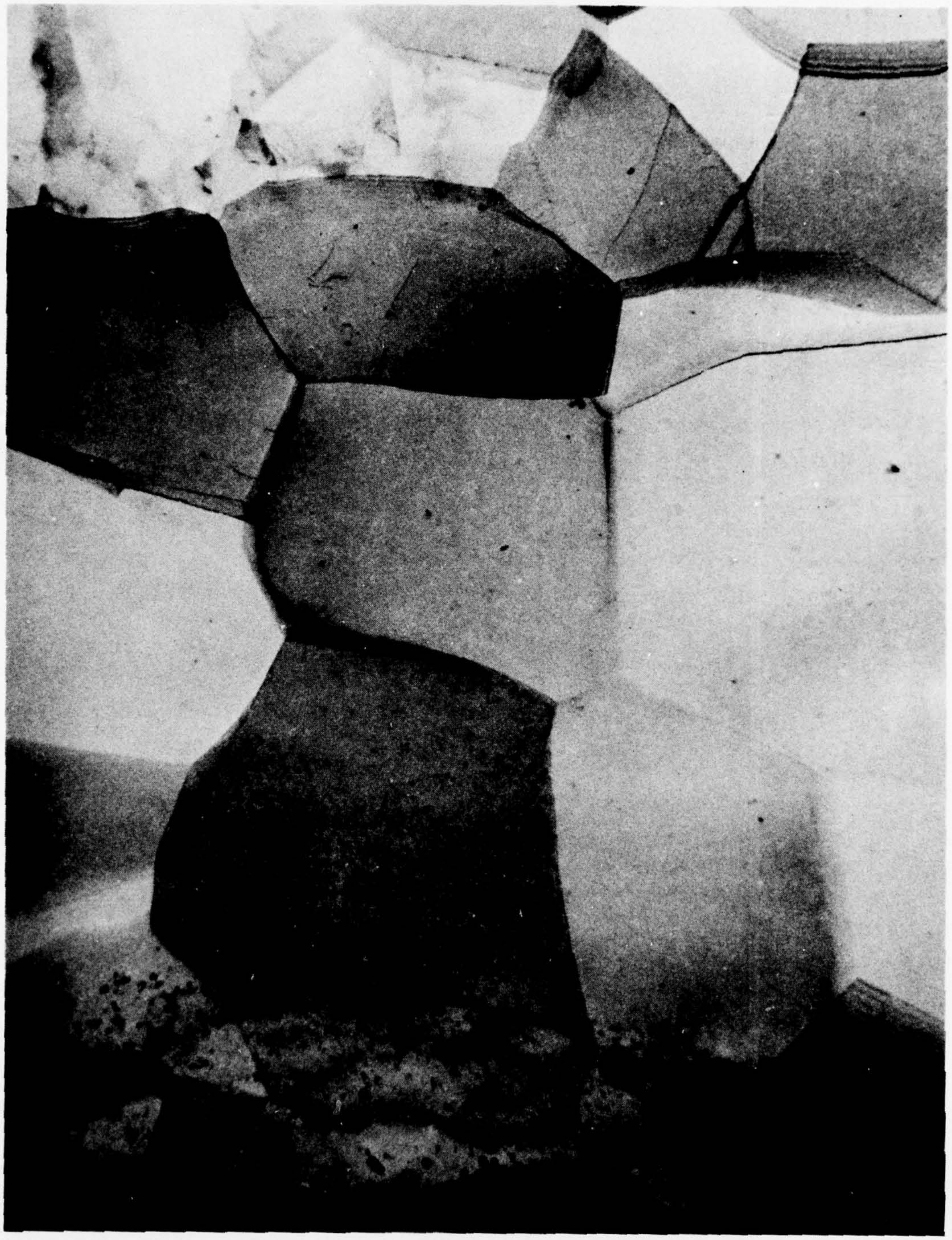

Fig. 19 Transmission Electron Micrograph of High-Purity Powder Source Beryllium Alloy BOP 32, Upset Forged, Rolled 75% and Annealed 977 K 10 Minutes. Structure shows a mixture of fine recrystallized grains and subgrains. Magnification 14,000×

Section 6
CONCLUSIONS

(1) The impact toughness of high-purity, low-oxide, powder-source beryllium can be increased by thermomechanical treatments to a level not far below that of Al 7075-T6.

(2) Optimum toughness occurs when the microstructure consists of very fine (2 to 4 μm) recrystallized grains in a matrix of subgrains which are on the threshold of recrystallizing in situ.

(3) There is a grain size controlled fracture mode change in both powder source and ingot source beryllium. The change is displaced to finer grain sizes by oxide particles and texture.

(4) To produce the finest grain size in beryllium for a given total reduction, a thermomechanical process should avoid any recrystallization before the final annealing treatment.

Section 7
FUTURE WORK

Most of the work reported here has been on textured beryllium products. In the remainder of this contract, work on nontextured upset forged products will be conducted to determine ductility and toughness as a function of grain size, degree of recrystallization, and the fracture and flow mechanisms involved.

Section 8
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